UDC 661.862.2 : 620.1

Ref. No. : ISO/R 900-1968 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 900

ALUMINIUM OXIDE

PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF TITANIUM CONTENT

Tiron photometric method

1st EDITION
December 1968

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 900, *Aluminium oxide primarily used for the production of of aluminium – Determination of titanium content – Tiron photometric method,* was drawn up by Technical Committee ISO/TC 47, *Chemistry,* the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question by the Technical Committee began in 1962 and led, in 1966, to the adoption of a Draft ISO Recommendation.

In May 1967, this Draft ISO Recommendation (No. 1170) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Israel	Spain
Belgium	Italy	South Africa, Rep. of
Bulgaria	Japan	Sweden
Canada	Korea, Dem. P. Rep. of	Switzerland
Czechoslovakia	Korea, Rep. of	Thailand
France	Netherlands	Turkey
Germany	New Zealand	U.A.R.
Hungary	Norway	United Kingdom
India	Poland	U.S.A.
Iran	Portugal	U.S.S.R.
Ireland	Romania	Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in December 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 900 December 1968

ALUMINIUM OXIDE

PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF TITANIUM CONTENT

Tiron photometric method

1. SCOPE

This ISO Recommendation describes a tiron photometric method for the determination of titanium in aluminium oxide primarily used for the production of aluminium.

2. FIELD OF APPLICATION

The method is applicable to the titanium contents found in the industrial material, providing that the Fe_2O_3 content is less than 0.045 % and the V_2O_5 content less than 0.008 %.

2.1 Special cases

2.1.1 When the Fe_2O_3 content is greater than 0.045 % (see clause 8.1).

2.1.2 When the V_2O_5 content is greater than 0.008 % (see clause 8.2).

3. PRINCIPLE

Formation of the titanium-tiron complex at pH 3.8 ± 0.1.

Photometric measurement of the coloured complex at a wavelength of about 380 nm.

4. REAGENTS

Distilled water or water of equivalent purity should be used in the test.

4.1 *Sodium carbonate*, anhydrous.

4.2 *Boric acid* (H_3BO_3).

4.3 *Nitric acid*, approximately 8 N solution.
Dilute 540 ml of nitric acid solution d = 1.4 (approximately 68 % m/m solution) with water to 1000 ml.

4.4 *Tiron*, 80 g/l solution.
Dissolve 80 g of tiron (disodium -1.2 dihydroxy -3.5 disulphonate) in water and dilute to 1000 ml. The quality of the solution of this reagent should be confirmed by a preliminary test. The solution should be prepared at weekly intervals.

4.5 *Sodium acetate*, approximately 4 M solution.
Dissolve 54.4 g of sodium acetate trihydrate in water and dilute to 100 ml.

4.6 *Buffer solution* (pH 3.8).
Dissolve 98 g of sodium acetate trihydrate in about 400 ml of water, add 280 ml of glacial acetic acid d = 1.05 (approximately 17.4 N solution) and dilute to 1000 ml.

6. PROCEDURE

6.1 Preparation of the sample solution (principal solution P).

Follow the instructions given in clauses 5.1, 5.2 and 5.3 of ISO Recommendation R 804, *Aluminium oxide primarily used for the production of aluminium – Preparation of sample solution for analysis,* making the final volume of the principal solution 250 ml.

This sample solution should be prepared just before use.

6.2 Test portion

Into both a beaker of convenient size (100 ml, for example) and a 100 ml one-mark volumetric flask transfer a 25.0 ml aliquot of the principal solution (6.1), each containing 0.500 g of dried* sample.

6.3 Preparation of the calibration curve

6.3.1 *Preliminary test to adjust pH.* Transfer to a beaker of convenient size (100 ml, for example) 8 ml of the standard titanium solution (4.10) containing 32 µg of TiO_2 and 2 ml of ascorbic acid solution (4.7) and dilute to approximately 40 ml. Add 10 ml of the tiron solution (4.4) (see clause 9.1) and then add slowly, by means of a graduated pipette or a burette, stirring after each addition, the volume of sodium acetate solution (4.5) required to adjust the pH to 3.8 ± 0.1.

Measure the pH value of the solution with the pH meter (5.1).

Note the volume of sodium acetate solution required to adjust the pH and discard this solution.

6.3.2 *Preparation of the standard matching solution* for photometric measurements in a 3 cm cell.

6.3.2.1 *Taking aliquot parts of the standard solution.* Into each of a series of eight 100 ml one-mark volumetric flasks transfer the volumes of the titanium standard solution (4.10) indicated by the following table and dilute each to about 40 ml.

Titanium standard solution (4.10)	Corresponding to TiO_2
ml	µg
0[1]	0
2.0	8
4.0	16
6.0	24
8.0	32
10.0	40
12.0	48
15.0	60

(1) Compensation solution

6.3.2.2 *Development of the colour.* Add to each flask 10 ml of the tiron solution (4.4), 2 ml of the ascorbic acid solution (4.7) and then the volume of the sodium acetate solution (4.5) used for the adjustment of pH in the preliminary test (see clause 6.3.1). Add 30 ml of the buffer solution (4.6), dilute to the mark and mix.

6.3.3 *Photometric measurement.* After 20 minutes, carry out the photometric measurement using either a spectrophotometer (5.2) at a wavelength of about 380 nm or a photoelectric absorptiometer (5.3) with a suitable filter, adjusting the instrument to zero optical density using the compensation solution (see clause 9.2).

6.3.4 *Preparation of the calibration chart.* Prepare a calibration chart having, for example, the titanium content in milligrammes of titanium dioxide (TiO_2) per 100 ml of solution, as abscisse and the corresponding values of optical density as ordinates.

* See ISO Recommendation R 802, *Aluminium oxide primarily used for the production of aluminium, Preparation and storage of test samples,* clause 2.3.